IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Appl. No. : 10/659,577 Confirmation No. 4666

Applicant : Lawrence T. Drzal and Hiroyuki Fukushima

Title : EXPANDED GRAPHITE AND PRODUCTS PRODUCED THEREFROM

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Examiner : Niland, Patrick Dennis

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Customer No. : 35684

MAIL STOP AMENDMENT
Commissioner for Patents
P.O. Box 1450
Alexandria, VA 22313-1450

SUPPLEMENTAL DECLARATION UNDER 37 C.F.R. § 1.132

Dear Sir:

Lawrence T. Drzal and Hiroyuki Fukushima, both inventors and Applicants in the above referenced application, state as follows:

(1.) That in the Declaration Under 37 C.F.R. § 1.132, filed April 27, 2009, in paragraph 8, it was stated that the microwave treatment of Sample B produced a surface area of 55 m^2/g.

(2.) That by contrast, Sample A, which was heated to 1000 °C by conventional heating, produced a surface area of 33 m^2/g.

(3.) That Sample A and Sample B were the same type of graphite, Asbury Expandable Graphite Grade 3772.

(4.) That the degree of expansion is usually expressed by the times expansion of the unexpanded graphite. The degree of expansion produced by the claimed invention with microwave or radiofrequency heating is greater than that produced by a conventional heating process at 600 °C to 1200 °C for the same graphite material. The resulting expanded graphite samples are distinctly different for microwave treatment than for conventional heat treatment.

(5.) In order to quantify the results, the expansion was evaluated by the BET surface area, which were set forth in the previous Declaration Under 37 C.F.R. § 1.132 dated April 27, 2009 as stated above in paragraphs (1) and (2). The pore structure of the said samples was characterized by measuring the N_2 adsorption-desorption isotherms on a Coulter Omnisorp 360 CX Sorptometer at 195°C using standard continuous sorption procedures. Before the measurement, each sample was heated overnight at 150°C and 10^{-6} Torr. The specific surface area ($S_{BET}[m^2/g]$) and the total pore volume (V_r [cc/g]) were calculated from the isotherms following the IUPAC recommendation (Sing et al., Pure Appl. Chem., 57, 603-619 (1985)). The attached Table A1 (See Exhibit A) from the previous Declaration Under 37 C.F.R. § 1.132 filed June 21, 2006 (shown as Table 2.1), shows the difference in the surface area with microwave heating to exfoliate the graphite. The surface area of the microwave exfoliated graphite was more than 4 times greater than graphite treated by direct application of heat to 600 °C to 1,200 °C as set forth on page

3, paragraph [0007] of the specification which was an unexpected result.

(6.) That the surface area can be directly correlated to the degree of expansion as shown in Figure B1 (See Exhibit B). Figure B1 shows the correlation between surface area and degree of expansion. Moreover, the degree of expansion and thus surface areas achievable by microwave treatment cannot be achieved through conventional heating for the same graphite material.

(7.) That the degree of expansion was defined as follows:

$$\text{Degree of Expansion} = \frac{\text{Volume after Expansion}}{\text{Volume before Expansion}}$$

(8.) That the volume before expansion was determined by dividing the weight of the graphite sample with the bulk density of graphite. The bulk density of graphite depends on the shape and size of the sample. The bulk density of the raw graphite sample used was determined by the volume occupied with the weight of the graphite sample, which was 1 ml/g.

(9.) That two sets of GICs (graphite intercalated compounds) were used in the experiments of Figure B1. One is the GIC made by Michigan State University and the other is a commercially available GIC from Asbury Carbon (Expandable Graphite A3772). All the MSU GIC samples were made from the same raw material by using the same process conditions. The results of the microwave treatment are shown in blue dots. This illustrates a clear correlation of surface area versus degree of expansion. This further shows that it is possible to achieve expansion of 300 times or more using microwaves. The degree of expansion depends on the starting graphite

material and power of the microwave. Regardless of the graphite used or the power of the microwave, microwave heating expands graphite greater than treating with conventional heating.

(10.) That the A3772 sample was expanded by microwave (1200W, 15 seconds) and is shown as a red dot. The A3772 sample was also heat treated in a conventional oven (800°C, 30seconds) and is shown as a Pink dot. That the degree of expansion for the microwave heating is greater than that of the conventional heating.

(11.) That the microwave treatment of the sample (red dot) resulted in a surface area over 50 m^2/g (similar to Sample A of the previous Declaration) which correlates to an expansion of approximately 240 times. The heat treatment sample shown in a pink dot resulted in a surface area of approximately 30 m^2/g (similar to Sample B of the previous Declaration). Accordingly, heat treatment did not achieve a surface area and thus a degree of expansion as that of microwave treatment for the same sample.

(12.) That heat treating intercalated graphite by conventional heat of 600 °C to 1,200 °C cannot achieve an expansion of 300 times or more. This is shown by the pink dog in the A3772 sample as well as the correlated conventional heat treated samples from Table A1 which achieved surface areas of 10.5 and 24 m^2/g. These conventional heat treated samples correlate to a degree of expansion of under 100 times with respect to Figure B1.

(13.) That the undersigned declares further that all statements made herein of his own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issuing thereon.

Lawrence T. Drzal

Lawrence T. Drzal

Date: 11/02/09

Hiroyuki Fukushima

Hiroyuki Fukushima

Date: 11/02/09

EXHIBIT A

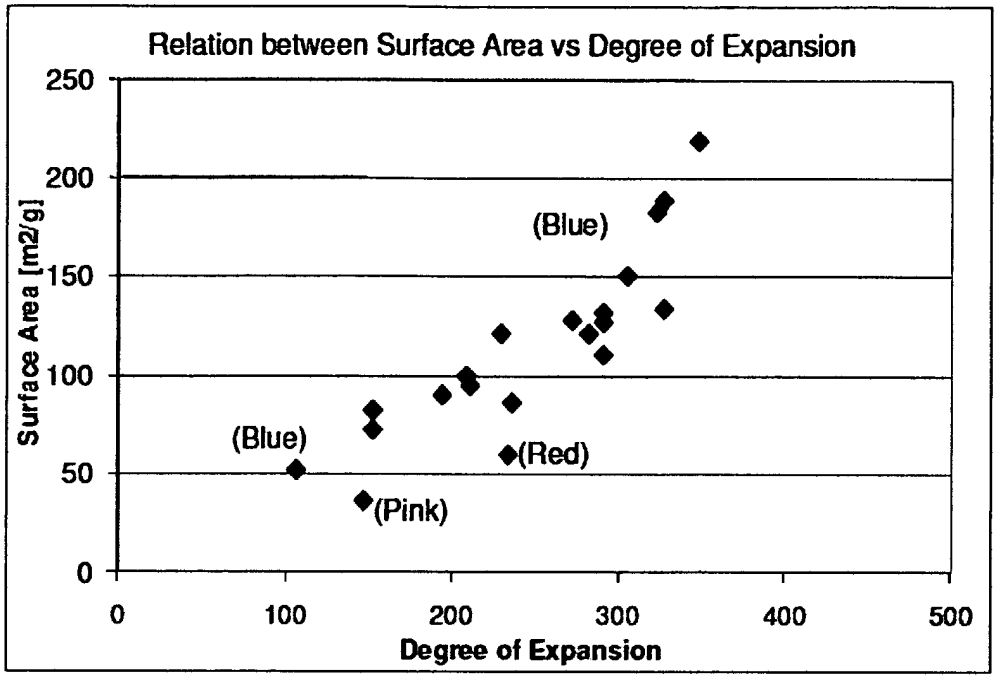
Table A1

Table 2.1. Thickness and Aspect Ratio of Graphite Samples Calculated from BET Surface Area Data

Sample	Surface Area (m^2/g)	Diameter (um)	Thickness (nm)	Aspect Ratio
As-received 160-50A	0.2	300	5172.4	58.0
Heat Exfoliated Graphite	10.5	15	96.5	155.5
Heat Milled Graphite	24	1.1	45.1	24.4
MW Exfoliated Graphite	105	15	9.5	1573.0
MW Milled Graphite	94	0.86	10.9	78.8

EXHIBIT B

FIGURE B1



Blue Dots = Microwave treatment of MSU GIC Samples.
Red Dot = Microwave treatment of Asbury Carbon Expandable Graphite A3772
Pink Dot = Conventional heat treatment of Asbury Carbon Expandable Graphite A3772